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L12: Entry 2 of 2 File: USPT Jan 18, 1994

DOCUMENT-IDENTIFIER: US 5279739 A
TITLE: Durable filtration membrane having optimized molecular weight

ABPL:
The subject of this invention is a novel polymeric composition useful in membrane technology such as microfiltration, nanofiltration, ultrafiltration, reverse osmosis and gas separation. The composition is formed by taking a known condensation polymer such as polyether sulfone, polysulfone or a polyarylether sulfone and modifying it to change the molecular weight distribution. The useful polymers of the invention will have an unmodified number average molecular weight of about 63,000 or less and weight fraction of molecules with a molecular weight of 50,000 or less in the range of 30 to 35%. Utilizing fractional precipitation or other known techniques for concentrating a high molecular weight fraction of a polymer the polymers are modified to have a number average molecular weight of a least 59,000 or a weight fraction of molecules with a molecular weight of 50,000 or less of not more than 19%. Superior resistance to cracking and other forms of membrane degradation results from using the polymer compositions of the present invention in filtration applications.

BSPR:
Known materials for use in forming ultrafiltration membranes include many commercially available polymers such as polyether sulfone, polysulfone, polyarylether sulfones, polyvinylidene fluoride, polyvinyl chloride, polyketones, polyether ketones, polytetrafluoro ethylene, polypropylene and polyamides. The foregoing polymers differ widely in their physical properties and the particular material selected is based upon the properties necessary to support a particular use. While the higher molecular weight "chain" polymers such as polyvinyl chloride and polytetrafluoro ethylene exhibit superior resistance to degradation from cleaning chemicals, they also have undesirable attributes which eliminate them from consideration as membrane materials for certain applications. On the other hand, the lower molecular weight "condensation" polymers which exhibit certain desirable properties particularly suited for ultrafiltration applications are not as durable and have a short and unpredictable service life when exposed to the types of chemicals aforementioned.

DEPR:
Suitable condensation polymers include polyether sulfone, polysulfone and polyarylether sulfones. Polyether sulfone is represented by the formula: ##STR1##

DEPR:
Polysulfones useful in the present invention are represented by the formula: ##STR2##

DEPR:
Suitable polysulfones for carrying out the present invention will have an MN of 43,000 to 54,000 and a WF=19.8 to 23%.

DEPR:
The modified polymer blended with PVP was dissolved in a mixture containing N-methyl pyrrolidone and sulfolane. A suitable pore former, such as ethylene glycol or lithium chloride, both well known to those skilled in the art, was added in a quantity to present a concentration of from 1 to about 10 % (w/v) of the pore former in the final product. The amount of pore former added was determined by the degree of porosity desired in the final product. The admixture was thoroughly blended and after degassing using conventional techniques was cast on a substrate such as a nonwoven fabric. A doctor blade was employed to disperse the casting in a uniform manner at a thickness of approximately 10 mils. The smoothed product was immediately gelled by immersion in a bath of cold water and N-methyl pyrrolidone [NMP being present from 0-70% (v/v)] for 15 seconds followed by continuous water washing for approximately 24 hours to remove all extraneous extractable contaminates.

DEPR:
While the invention encompasses any condensation polymer that capable of being formed into an ultrafiltration membrane, a preferred group of polymers is that consisting of polysulfone, polyether sulfone and polyarylsulfones such as poly(oxy-1,4-phenylene sulfonyl-1,4-phenyleneoxy-4,4'-biphenylene) and poly(oxy-1,4-phenylene sulfonyl-1,4-phenyleneoxy-4,4'-biphenyleneoxy-1,4-phenylene sulfonyl-1,4-phenyleneoxy-1,4-phenylene). The most preferred polymer for use in the present invention is polyether sulfone.

DEPR:
The hardened membrane material may be processed in a conventional matter to form the final membrane including the use of pore formers or to achieve the desired end product. Suitable pore formers include low molecular weight organic compounds, inorganic salts and organic polymers, for example vinyl pyrrolidone/dimethyl aminomethyl methacrylate; polyoxazolines such as poly(2-ethyl-I-oxazoline) and poly(2-methyl-2-oxazoline); copolymers of polysulfone such as polysulfone-b-polyethylene oxide and polysulfone-b-polyvinyl pyrrolidone; and copolymers of polyether sulfone such as polyether sulfone-b-polyethylene oxide and polyether sulfone-b-polyvinyl pyrrolidone.

DEPR:
Other suitable pore formers include low molecular weight organic acids such as acetic acid, propionic acid and sulfolane and inorganic salts such as lithium chloride, lithium bromide, lithium fluoride, sodium bicarbonate, sodium carbonate and sodium acetate. Organic polymers such as poly(N-vinyl pyrrolidone) and poly(ethylene glycol) may also be used as pore formers.

CLPR:
11. An ultrafiltration membrane as set forth in claim 7, wherein said condensation polymer is a member of the group consisting of polysulfone, polyether sulfone and polyarylether sulfones.

CIOR:
210/500.41

CCOR:
210/500.41

CIFS:
210/500.41

UROR:
210/500.41

UROR:
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UROR:
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UROR:
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FROR:
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FROR:
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L10: Entry 1 of 30 File: USPT May 8, 2001

DOCUMENT-IDENTIFIER: US 6228970 B1
TITLE: Poly (biphenyl ether sulfone)

DEPR:
The poly (biphenyl ether sulfones) of this invention have a higher number average molecular weight (Mn) for a given weight average molecular weight (Mw) compared to prior poly (biphenyl ether sulfone) materials. This provides for improved mechanical properties such as improved tensile strength, particularly for membranes and fibers. Preferably, the poly (biphenyl ether sulfones) of this invention have an Mn which meets the following equation:

DEPR:
The poly (biphenyl ether sulfones) of this invention have a relatively low polydispersity (i.e., Mw/Mn) compared to prior poly (biphenyl ether sulfone) materials. This provides for improved mechanical properties such as improved tensile strength, particularly for membranes and fibers. Higher amounts of low molecular weight oligomers, including cyclic oligomers, lowers the number average molecular weight whereas the weight average molecular weight is less affected. Thus where the level of low molecular weight oligomers is increased, the polydispersity is increased. Preferably, the poly (biphenyl ether sulfones) of this invention have a polydispersity which meets the following equation:

DEPR:
In the carbonate method, the polysulfones are prepared by contacting substantially equimolar amounts of 4,4'-biphenol, optionally with one or more other bishydroxy aromatic compounds such as 4,4'-dihydroxydiphenyl sulfone, hydroquinone, bisphenol A, and the like, and dihalodiarylsulfones, e.g., 4,4'-dichlorodiphenyl sulfone or 4,4'-difluorodiphenyl sulfone, with from about 0.5 to about 1.0 mole of an alkali metal carbonate per mole of hydroxyl group in a solvent mixture comprising a solvent which forms an azeotrope with water in order to maintain the reaction medium at substantially anhydrous conditions during the polymerization. The temperature of the reaction mixture is kept at about 170.degree. C. to about 350.degree. C., preferably from about 210.degree. C. to about 300.degree. C. for about one to 15 hours. Typically, if the reaction is conducted at atmospheric pressure, the temperature of the reaction is typically limited by the boiling temperature of the solvent selected.

DEPR:
In order to prepare such polyethersulfones having low levels of oligomeric materials, improved polydispersity and improved melt flow characteristics, the concentration of bisphenol S (and other bisphenols, if used) and the 4,4'-dihalodiphenyl sulfone is adjusted such that the percent solids (as defined above) in the polymerization reaction solution used to prepare the polysulfone is greater than about 35 weight percent, more preferably at least about 40 weight percent and most preferably at least about 50 weight percent. The preferred polar aprotic solvent used to prepare polyethersulfones made with a bisphenol comprising bisphenol S is sulfolane. Various amounts of at least one other bisphenol can be included with the bisphenol S, such as bisphenol A (4,4'-isopropylidenediphenol) or hydroquinone. If used, the amount of such other bisphenol is suitably about 1 to about 50 mole percent, preferably about 20 to about 30 mole percent of the total number of moles of bisphenol present. Preferably the other bisphenol if used is hydroquinone.

DEPR:
The improved polyethersulfones of this invention and useful for making a variety of articles such as films, sheets and fibers. They are particularly suitable for manufacturing molded articles such as electrical "chip" trays and other parts for electronic or electrical components as well as, for example, automotive fuses and membranes.

DEPL:
in tetrahydrothiophene oxides and dioxides. Specifically, these solvents include dimethylsulfoxide, dimethylsulfone, diphenylsulfone, diethylsulfoxide,

dimethylsulfoxide, dimethyls[redacted]one, diphenylsulfone, diethyls[redacted]foxide, diethylsulfone, diisopropyls[redacted]one, tetrahydrothiophene-1,1-d[redacted]ide (commonly called tetramethylene sulfone or sulfolane) and tetrahydrothiophene-1-monoxide. Additionally, nitrogen containing solvents may be used. These include dimethylacetamide, dimethylformamide and N-methyl-pyrrolidone.

ORPL:
Russian Article, Study of a Low-Molecular-Weight Cyclic Product in Polysulfone; Vysokomol. Soyedin., Ser. B., vol. 24, No. 1, pp. 66-619, 1982. Plastmassy Pilot Plant, the month in the date of publication is not available.